

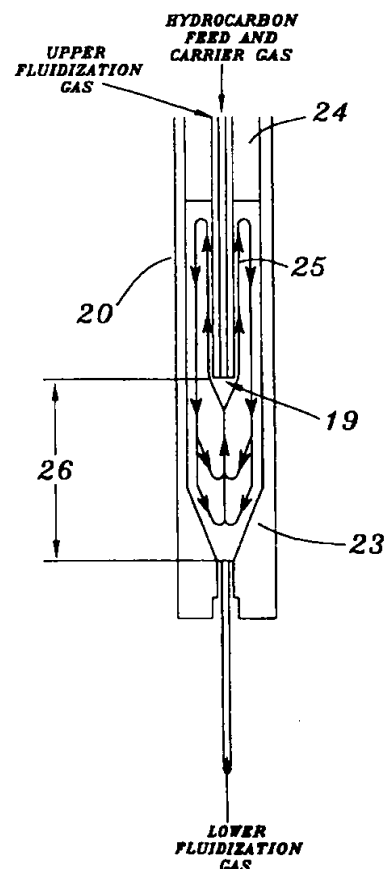
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International Bureau

## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification <sup>6</sup> : B01J 19/00		A1	(11) International Publication Number: WO 98/52685 (43) International Publication Date: 26 November 1998 (26.11.98)
(21) International Application Number: PCT/US98/11223 (22) International Filing Date: 22 May 1998 (22.05.98)  (30) Priority Data: 08/862,657 23 May 1997 (23.05.97) US  (71) Applicant: KAYSER TECHNOLOGY, INC. [US/US]; 15502 Oakmont Club Court, Houston, TX 77059 (US). (72) Inventor: KAYSER, John, C.; 4407 Walnut Pond Drive, Houston, TX 77059 (US). (74) Agent: MASON, Dwayne, L.; Matthews, Joseph, Shaddox & Mason, L.L.P., P.O. Box 572957, Houston, TX 77257-2957 (US).			(81) Designated States: CA, CN, JP, European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).  Published With international search report.

**(54) Title:** VERSATILE FLUIDIZED BED REACTOR**(57) Abstract**

A laboratory scale fluid catalytic cracking apparatus and method of use thereof, which provides cracking performance that emulates commercial riser cracking. The apparatus includes a reactor (20) having a removable feed injector (24) to quickly facilitate changing hydrocarbon contact time without varying the feed rate or diluent rates, or catalyst charge, and also without the expense of a new reactor. The feed injector is a tube (24) within a tube design. The feed injector allows hydrocarbon feed as well as fluidization gas to be delivered to a prescribed axial position within a catalyst bed in the reactor to directly affect hydrocarbon contact time. The reactor also includes a conical bottom head (23) having a conical section and a lower fluidization gas nozzle connected at its apex. The total included angle of the conical section may vary between 10 and 170 degrees. The reactor geometry combined with the location of the fluid and gas sources generate the desired torrodial motion of the catalyst bed, which is significantly enhanced over conventional designs which do not use multiple nozzles and the conical bottom design.



## Description

### VERSATILE FLUIDIZED BED REACTOR

#### TECHNICAL FIELD

The present invention relates to the design of a fluidized bed reactor one use of which is for laboratory evaluation of the fluid catalytic cracking process with particular regard to catalysts, feedstocks, and process parameters. Fluid catalytic cracking is the dominant catalytic process for producing transportation fuels and chemical feedstocks worldwide. Consequently, extensive efforts have been made at developing useful laboratory tests pertinent to this process for the purposes of developing improved catalysts, quantifying and correlating the cracking character of various feedstocks based on their respective properties, understanding the implications of different process conditions, and improving commercial process design. The present invention also relates to the design of fluidized bed reactors, which have applications in the hydrocarbon and chemical process industries on commercial, pilot plant, and laboratory scales.

#### BACKGROUND

The two broad approaches commonly used in the laboratory for studying the fluid catalytic cracking process are continuous processing units and batch processing units. The continuous processing units are basically scaled-down versions of commercial operating units and are typically very complex systems that are expensive to construct, operate, and maintain. In addition, they require large samples of catalyst as well as feed compared to

than that used when the contact time is longer. It is therefore important for the laboratory scale fluidized catalytic cracking apparatus to provide the flexibility to vary contact time and simultaneously operate at high catalyst-to-oil ratios.

5        There are several ways to vary contact time in laboratory fluid-bed reactors. The widely known techniques of altering the hydrocarbon feed rate, the rates of any diluent gases, and/or altering the catalyst charge provide results, however, the results are not entirely consistent with commercial experience or have other deficiencies which limit their applicability.

10        Walsh, U.S. Pat. No. 4,419,328, discloses a laboratory apparatus for investigating the performance of catalytic cracking catalyst utilizing batch techniques and a fluidized bed reactor. Walsh teaches the use of the laboratory apparatus and techniques for obtaining cracking data and not a reactor apparatus or method that can be utilized to emulate the performance  
15        characteristics expected in a commercial scale reactor. Walsh discloses a fluidized bed reactor but it does not include a movable feed injector or disclose in any way the important aspects pertaining to injector location and its relationship to controlling hydrocarbon contact time. Walsh does not disclose or teach the injection of multiple feeds at different locations. In  
20        addition, Walsh does not disclose or teach an apparatus or method to achieve the catalyst circulation pattern within the reactor including its relation to commercial catalytic cracking as well as ways to enhance the circulation by proper reactor design and injector positioning.

with the commercial scale FCCU. The present invention shows that moving the feed injector does not significantly alter the catalyst fluidization pattern as compared with altering the feed rate, and does not require any changes in the injection time to attain a prescribed catalyst-to-oil ratio.

5        Varying the diluent rates has the same effect on catalyst patterns as changing the feed rate. In addition, varying diluent rates changes the hydrocarbon partial pressure which affects yields and performance. Simply moving the feed injector does not affect hydrocarbon partial pressure.

10        Decreasing the catalyst load to decrease contact time reduces bed height and can result in a short bed within which it is more difficult to distribute the hydrocarbon feed thereby producing more erratic yields. In addition, as catalyst load is reduced it becomes difficult to measure yields at high catalyst-to-oil ratios which is inherently important to reduced contact time operation. This difficulty is because the feed mass is very low and difficult to account for  
15        in a mass balance (at high catalyst-to-oil ratio with reduced catalyst mass). In addition, at constant catalyst-to-oil ratio, varying the catalyst load to affect contact time also requires changing the feed injection time, which adds complication to the kinetic analysis and comparison of results to a commercial scale FCCU.

20        Another feature of the invention couples the reactor geometry with the gas and feed supply rates and injector locations to provide enhanced catalyst circulation up the center core of the reactor and down the walls. This pattern is natural to fluidized beds, but it is enhanced by the present invention and

relevant performance over a wider range of velocity and may allow for higher feed throughput without degradation in performance.

Another objective of the present invention is to provide a versatile fluidized bed reactor that emulates the yield performance of commercial fluid catalytic cracking units, which inject feed to be cracked at one or more feed locations in a nearly ideal plug flow riser reactor. Heretofore, either different equipment or a totally new reactor was necessary to accomplish the desired testing in the laboratory. Multiple feed locations are necessary since different feeds require different contact times to obtain optimal yields. The present invention accomplishes this in that contact time may be varied by the methods described herein utilizing the reactor of the present invention without modification to accommodate multiple feeds while producing commercially relevant results.

Another objective of the present invention is to provide a reactor design, which may be used in processes other than fluid catalytic cracking on any scale. For example, the methods and apparatus described herein may be used in catalyst deactivation of fluid catalytic cracking catalyst as well as for chemical reactions including but not limited to partial oxidation reactions, an example of which is oxidation of ethylene to produce ethylene oxide.

#### **BRIEF DESCRIPTION OF THE DRAWINGS**

FIG. 1 Is a schematic representation of a fluidized bed reactor.

FIG. 2 Is a detailed schematic representation of a fluidized bed reactor.

FIG. 3 Is a graphical representation of 430°F+ Conversion vs. Cat/Oil Ratio referenced in Example 1.

connected through the bottom head 23 aiming vertically upward into the catalyst bed 25. The combination of the reactor geometry and fluid injection results in the flow pattern represented by the arrows within the catalyst bed 25. One skilled in the art will recognize that the present invention may be practiced utilizing reactors employed in the laboratory, pilot plant, or commercial scale manufacturing.

Feed is injected into the reactor and directly contacts the catalyst bed 25. The center or core of the reactor is of lower density since it includes gas flows and vapor phase cracked products in addition to catalyst. These materials thus flow upward, as noted by the flow arrows shown within the catalyst bed 25 and almost all of the hydrocarbon vapor products escape out of the top of the catalyst bed while the catalyst circulates primarily back down along the wall of the reactor 20. The time that the hydrocarbons contact the catalyst is directly related to the distance from the top of the catalyst bed from which feed is injected. Therefore, by gauging the distance of the feed injector from the apex of the bottom head 23, illustrated by arrow 26, it is possible to systematically alter the reaction contact time.

Figure 2 is a more detailed schematic representation of a fluidized bed reactor according to the present invention. The fluidized bed reactor 20 shown in Figure 2 comprises a reactor shell 21 having a top head 22 and a bottom head 23 connected at either end of the shell forming a pressure vessel.

prescribed length may be installed in its place. The feed line 1 similarly may be a compression fitting 5 disposed about the exterior of the feed line 1 to seal against a seat within the upper end of the fluidization gas line 2. The feed line 1 may be removably retained by feed line coupling 6 together with the compression fitting 5 to forming a pressure tight seal. While the outlet or injection point of both the feed line 1 and the fluidization gas line 2 as shown in Figure 2 terminate at the same point within a catalyst bed 25, it is within the scope of the present invention that each may be terminated at different points within the catalyst bed 25. The feed injector 24 and fluidization gas line 2 are used to supply inert gas, such as nitrogen or steam, to the feed injection point, (upper arrow 26) to both prevent catalyst particles from entering and plugging the feed line 1 and to cool the feed injector 24. The feed injector 24 of Figure 2 is shown as a tube within a tube directed downward into the interior catalyst bed 25 of the reactor 20. However, the feed injector 24 may (as noted above) be connected through the bottom head 23 aiming vertically upward. An injector centering means 27 may be connected directly to the bottom surface of the reactor top head 22 or to the reactor shell 21. The centering means 27 provides a guide path for the feed injector 24 so that the injector 24 remains aligned with the axis of reactor 20.

The bottom head 23 includes a conical section 7 of the reactor 20. The bottom head 23 also includes a fluidization nozzle 8, connected at the bottom or apex of the conical section 7, through which a lower fluidization gas line 9 extends upward to provide fluidization gas, such as nitrogen or other diluent,

extends axially through the effluent product nozzle 14 providing a conduit for the flow of gases to other laboratory equipment (not shown). A compression fitting 16 is disposed about the exterior surface of the effluent product line 15 and seals against a seat within the outlet of the effluent product nozzle 14. A pressure tight seal is created by connecting effluent coupling 17 to the outlet of the effluent product nozzle. A product filter 18 may be connected at one end of the effluent product line 15 to prevent carry over of catalyst with the gases.

To prepare the reactor 20 for commercial simulation testing of a FCCU process, the appropriate feed injector 24 is first inserted through the top head 22 and the injector coupling 3 is connected to the top head 22 to establish a fluid tight seal. The feed injection point (designated by arrow 19) is now established with respect to the bottom of the catalyst bed, designated by lower arrow 26, and thus the catalyst-to-feed contact time is established for the prescribed catalyst charge and injection rates. The reactor 20 is then charged with the desired mass of catalyst through the catalyst nozzle 12 while fluidization gas is allowed to flow through the feed line 1, and the fluidization gas lines 2 and 9 into the catalyst bed 25. This results in the desired torodial motion of the catalyst bed 25. The reactor 20, catalyst bed 25, and fluidization gases are operated at a desired temperature via temperature control means (not shown). The temperature control means may be an external jacket type heating element or other means well known in the art.

Techniques and advantages over the prior art methods and apparatus are illustrated in the following non-limiting examples:

### EXAMPLE 1

To illustrate the operation, three feed injector locations were used to crack the feedstock described in Table 1 over the catalyst described in Table 2. The catalyst charge to the reactor is 9.0 gms and the cracking temperature is 990°F (532°C). The results are compared in Table 3 at a catalyst-to-oil ratio of 5.0 and some of the salient results are illustrated in Figures 3 through 7.

TABLE 1  
FEED PROPERTIES

Feedstock		Feed A
API Gravity		21.2
Specific Gravity, 60/60°F		0.927
Sulfur, wt%		0.87
Conradson Carbon Residue, wt%		0.8
Distillation (D 2887)		°F/°C
	wt%	
	10	659/348
	50	833/445
	90	983/528

TABLE 2  
CATALYST PROPERTIES

Catalyst	Equilibrium Cat.
Total SA, m <sup>2</sup> /gm	213
Zeolitic SA, m <sup>2</sup> /gm	142
Matrix SA, m <sup>2</sup> /gm	71
Z/M	2.0
RE <sub>2</sub> O <sub>3</sub> , wt%	2.4
UCS	24.34
Nickel, ppmw	1400
Vanadium, ppmw	2500

Decreasing contact time impacts conversion, coke, and delta coke as shown in Figures 3 through 5. At constant coke, as contact time decreases (going from the 1.125" injector to the 2.625" injector), conversion decreases, delta coke decreases, and required catalyst-to-oil increases. Hydrocarbon  
5 contact time influences dry gas and bottoms conversion as shown in Figures 6 and 7.

Table 3 shows all the yield shifts characteristic of decreasing contact time by comparing the data at constant catalyst-to-oil. Reducing contact time of commercial operations usually increases gasoline yield. However, as very  
10 short contact times are employed the gasoline yield begins to decrease because of conversion loss. This is effectively shown in another way at constant catalyst-to-oil ratio in Table 3.

#### EXAMPLE 2

To illustrate the relevance of the particular laboratory apparatus, data  
15 are provided in Table 4 comparing the laboratory unit to a particular commercial, full-scale fluid catalytic cracking operation with both the laboratory and commercial unit operating on the same feed and catalyst. In the Table the ratio of the laboratory yields to commercial yields are indicated and the closer the values are to unity the closer is the correspondence of the  
20 performance of the laboratory and commercial unit.

The commercial unit is a fully modernized short contact time unit which is operating well (with radial feed nozzles, good riser termination, and good stripper design). The operation of the invention involved tuning the

## EXAMPLE 3

To illustrate the precision of the laboratory apparatus, which is a direct measure of the stability and repeatability of the fluidized bed, yield data for repeated runs are provided in Table 5. These data are with the same feed  
5 and catalyst described in Tables 1 and 2 and a temperature of 990°F (532°C).

The relative error values of Table 5 indicate the invention and associated analytical equipment are very precise. The relative error is nominally 2% or less for each yield. Since the analytical equipment used for these measurements are no more precise than the data itself, it is clear that  
10 the invention provides extremely reproducible performance. This is an indication of the stability of both the catalyst and feed contacting and the catalyst circulation issues discussed further in Example 4.

constant and the feed injector is 1.125 inches from the reactor bottom. The injector diameter is 0.125 inches for Reactor A and 0.250 inches for Reactor B.

The comments in Table 6 may be summarized as follows (with due qualification for the catalyst material studied): Reactor A (invention) maintains a very stable fluidized bed up to 0.50 ft/sec and the stability begins to deteriorate at velocities where solids entrainment becomes a factor. Reactor B (and many other designs) begins to slug (as large bubbles form) at superficial velocities as low as 0.15 to 0.25 ft/sec. Reactor A provides stable fluidization over a superficial velocity range that is about twice that of Reactor B. The catalyst circulates within Reactor A several times per minute as noted in Case 5 of Table 6 (at 0.35 ft/sec superficial gas velocity).

15

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above the conical bottom section. As the injector is elevated above the conical section the fluidization pattern remains stable and circulates with the toroidal motion shown in Figure 1 at all higher levels.

Since the axial position of the injector is directly related to contact time,  
5 it is clearly possible to supply one feed at one location and a second feed at another. In this way, the apparatus could be used to perform studies at multiple feed locations pertinent to commercial catalytic cracking operations.

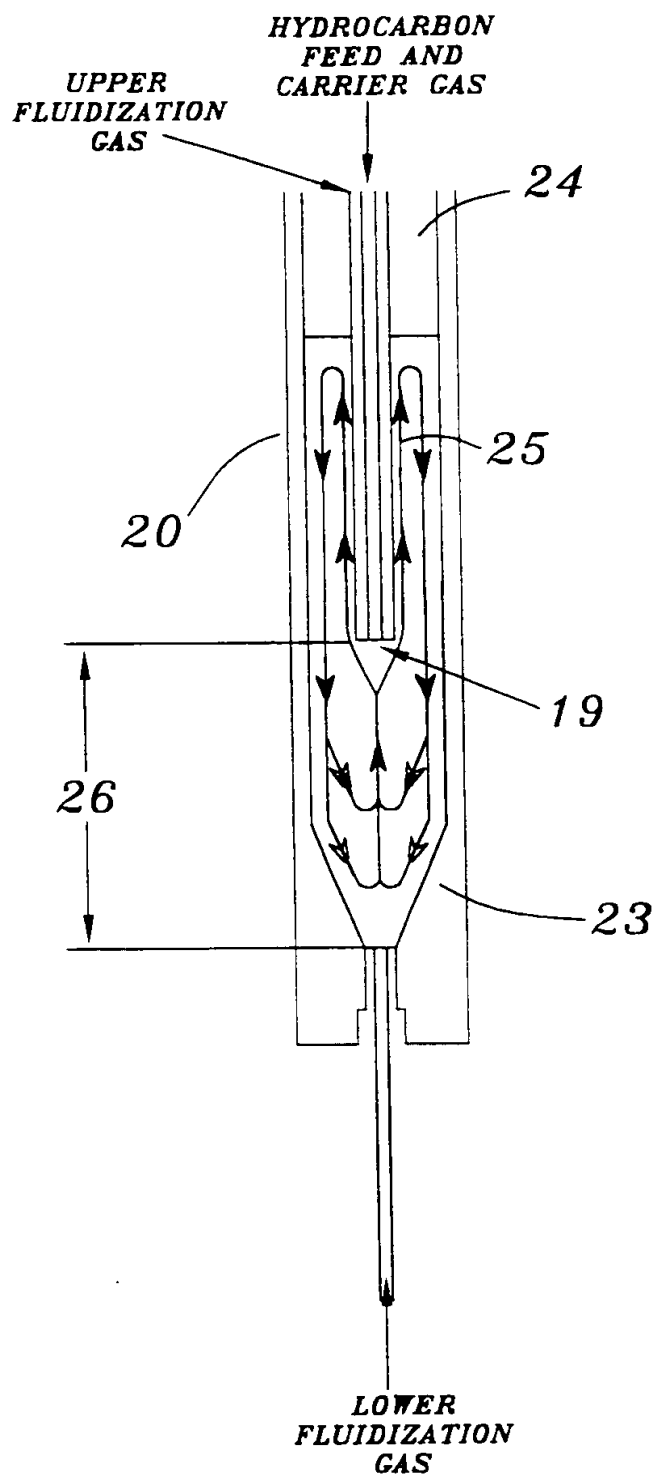
Moreover, while the particular embodiment of the invention has been shown specific to fluid catalytic cracking, modifications or application of the  
10 invention to other catalytic processes by someone skilled in the art of reactor design are within the spirit and scope of the invention. Since the laboratory apparatus works well at emulating continuous, short contact time fluid catalytic cracking operations, it will work well in other applications. For example, partial oxidation reactions like ethylene to ethylene oxide, and  
15 isomerization reactions could be performed in the type of fluid bed shown in Figure 1. In addition, there are many fluidized bed applications that may benefit from the invention where the solid is not strictly catalyst, the mobile phase is not strictly gaseous, and the process may not involve chemical reaction. Use of the invention in these applications is also considered within  
20 the spirit and scope of the invention.

In addition, also within the spirit and scope of the invention is continuous fluidized bed processing. Clearly, it is straightforward to apply the batch-wise concepts of this invention to fluid bed reactors operating in a

What is claimed is:

1. A versatile fluidized bed reactor apparatus, said apparatus comprising:  
a reactor comprising a reactor shell having a top head and a bottom  
head connected thereto; and  
5 wherein at least one of said heads having a removable feed injector  
extending axially there through for transmitting fluid at variable  
locations within said reactor.
2. The apparatus of claim 1, wherein said reactor further comprises a bed  
of solids material.
- 10 3. The apparatus of claim 2, wherein said solids material is a catalyst.
4. The apparatus of claim 1, wherein said feed injector comprises:  
an upper fluidization gas line; and  
a feed line connected to said outer tubular member and extending  
axially therethrough.
- 15 5. The apparatus of claim 1, wherein said feed injector extends axially  
through said top head.
6. The apparatus of claim 1, wherein said feed injector extends axially  
through said bottom head.
7. The apparatus of claim 1, wherein the conical bottom head comprises  
20 a conical section and a fluidization nozzle positioned proximate to an apex of  
said conical section.
8. The apparatus of claim 7, wherein a total included angle of said conical  
section is in the range of 10° to 170°.

- providing a source of heat for maintaining said vessel and said material bed at a desired temperature;
- charging said vessel with a mass of solids material to form a material bed within said vessel;
- 5 connecting said feed injector such that the outlet of said injector extends to a prescribed axial position within said material bed;
- and
- introducing a mass of fluid through said feed injector and into said material bed.
- 10 15. The method of claim 14, wherein said conical bottom head comprises a conical section and a nozzle connected at the apex of said conical section.
16. The apparatus of claim 15, wherein a total included angle of said conical section is in the range of 10° to 170°.
17. The apparatus of claim 15, wherein a total included angle of said
- 15 conical section is in the range of 20° to 60°.
18. The method of claim 14, wherein said feed injector comprises:
- an upper fluidization gas line; and
- a feed line connected to said outer tubular member and extending axially therethrough.
- 20 19. The method of claim 14, wherein said feed injector extends axially through said top head.
20. The method of claim 14, wherein said feed injector extends axially through said bottom head.

**FIGURE 1**

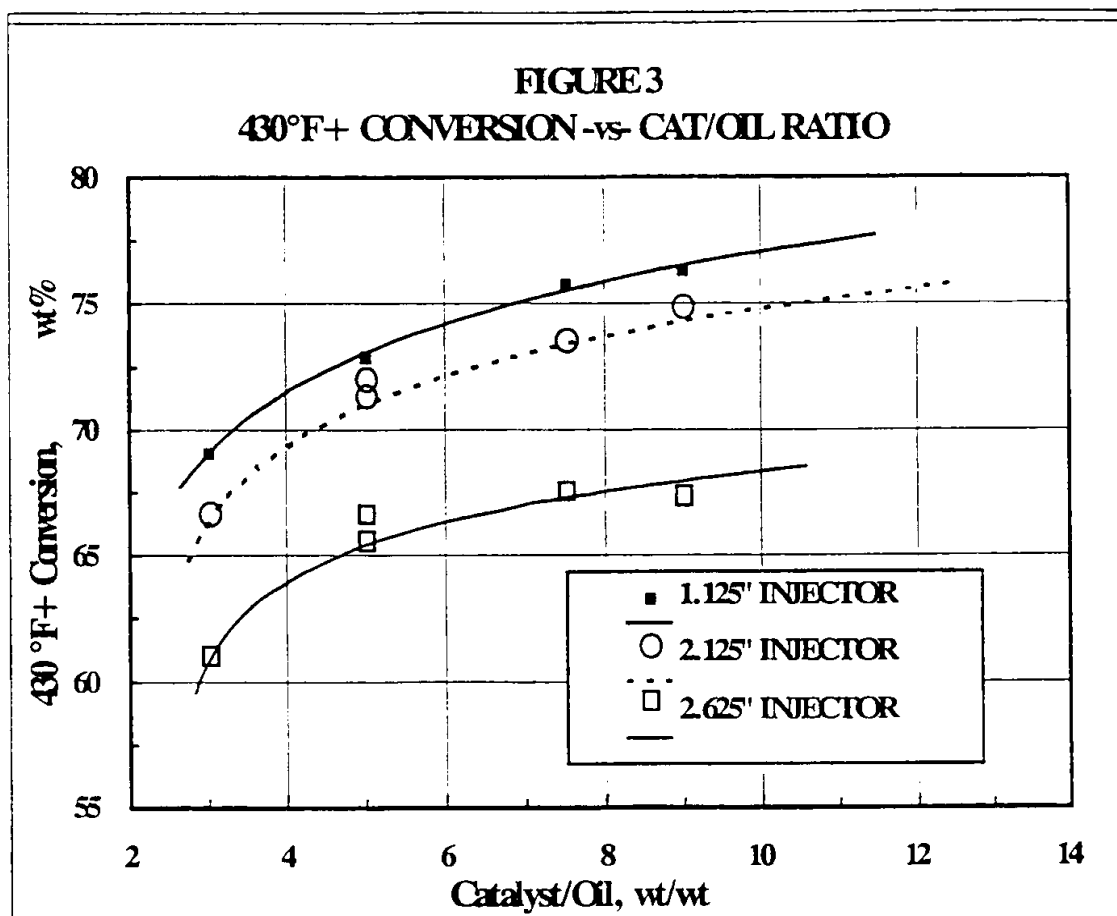
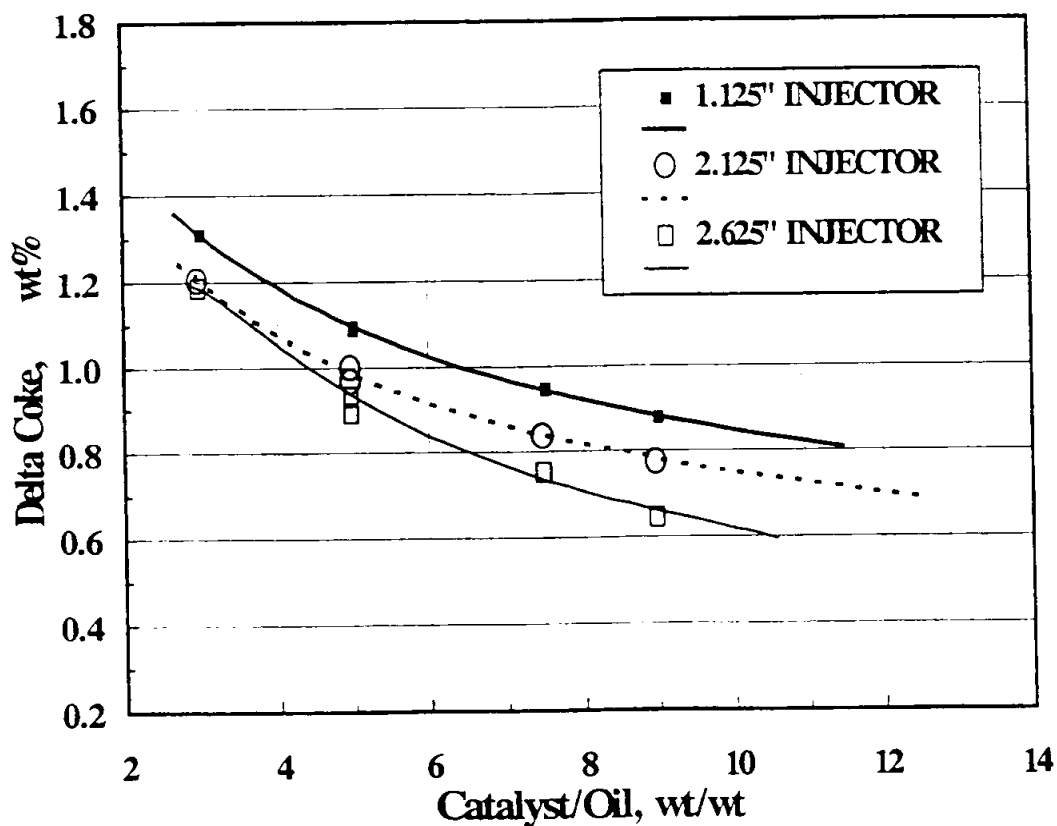
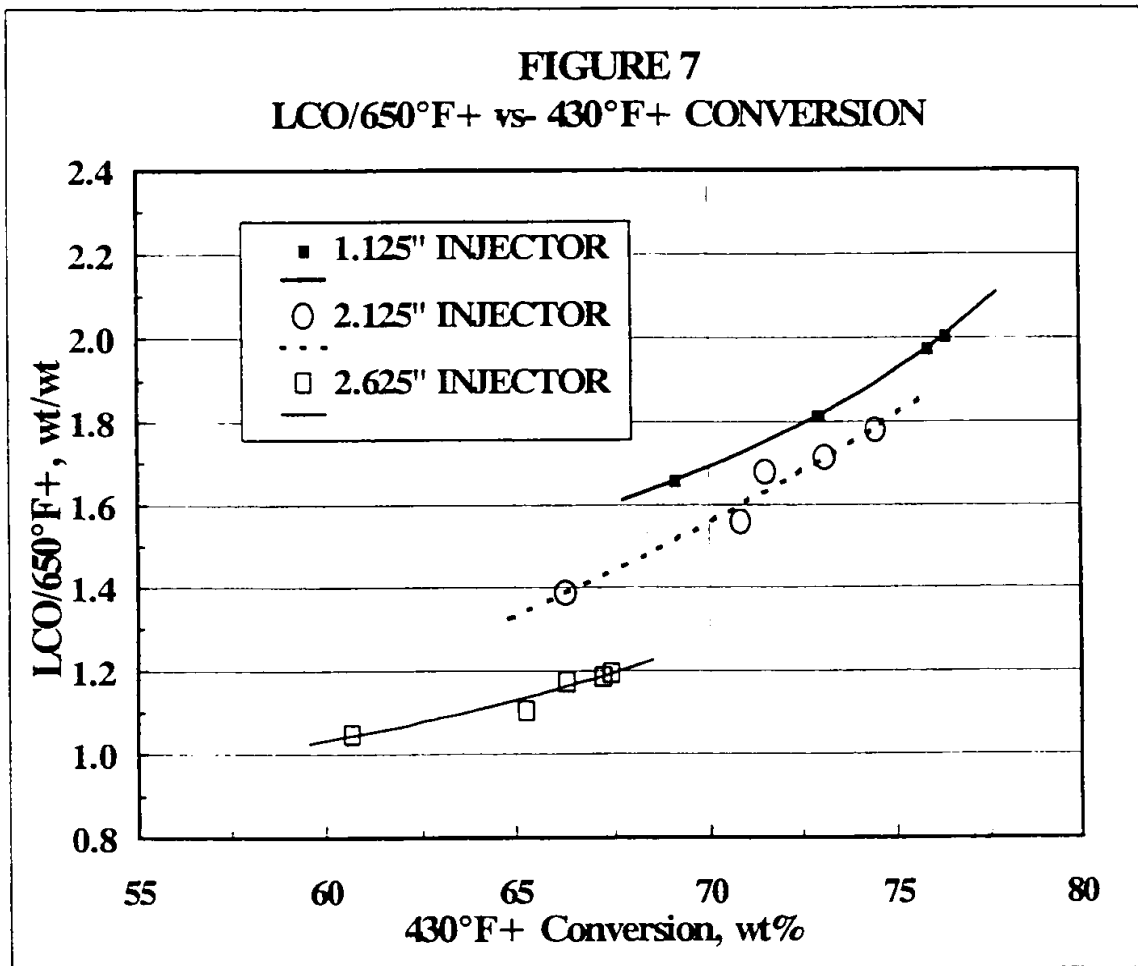


FIGURE 5  
DELTA COKE -vs- CATALYST/OIL





## INTERNATIONAL SEARCH REPORT

International application No.  
PCT/US98/11223

## A. CLASSIFICATION OF SUBJECT MATTER

IPC(6) : B01J 19/00

US CL : 422/130, 139; 436/37

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 422/130, 139; 436/37

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y ---- A	US 5,262,104 A (SCHWARTZ) 16 NOVEMBER 1993, see entire document.	1 - 3 , 5 - 17,19,20,25,26 ----- 4,18, 21-24



Further documents are listed in the continuation of Box C.



See patent family annex.

* Special categories of cited documents:	*T* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
*A* document defining the general state of the art which is not considered to be of particular relevance	*X* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
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Date of the actual completion of the international search

24 JUNE 1998

Date of mailing of the international search report

27 JUL 1998

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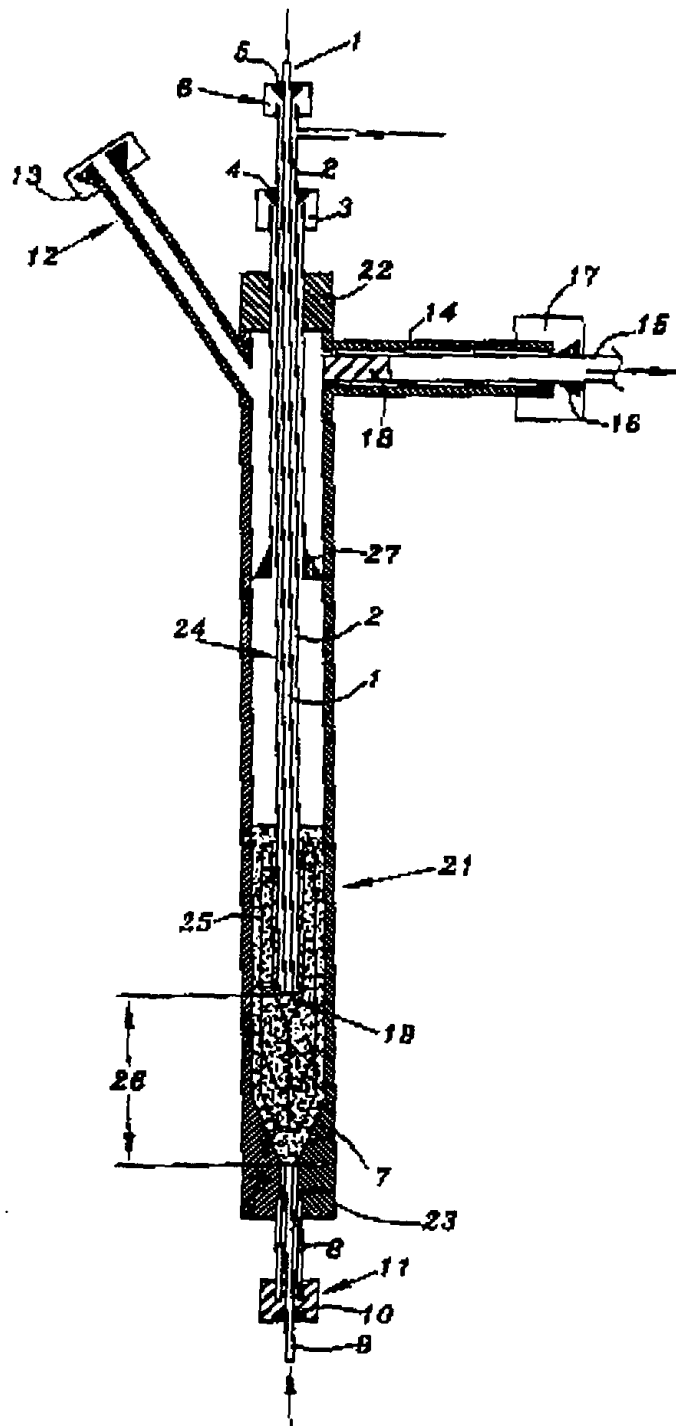


FIGURE 2

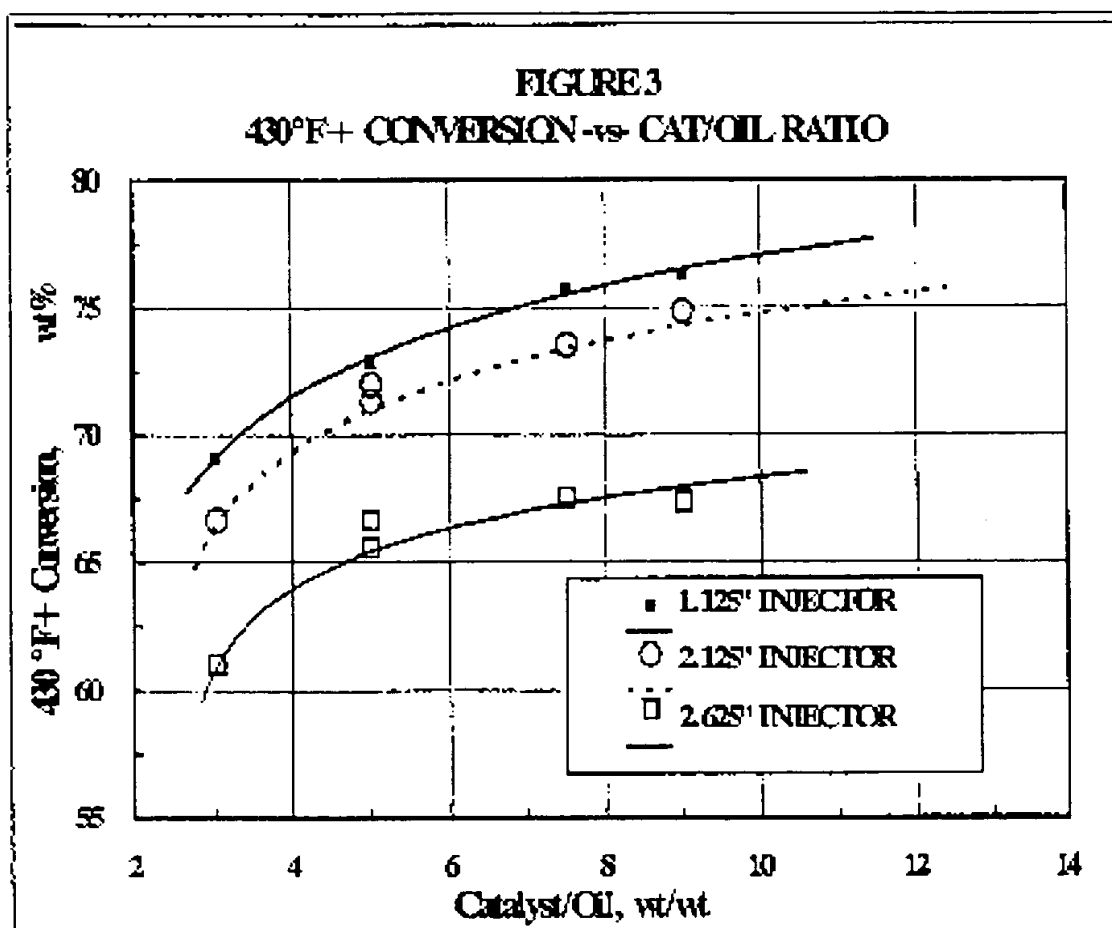


FIGURE 5  
DELTA COKE -vs- CATALYST/OIL

